Long-term Memory and Magnetoacoustic Effects at Excitation of Magnetostrictive Materials by RF and Magnetic Pulses Using Pulsed NMR Technique

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Abstract

Different long-term acoustic memory and magnetoacoustic effects were observed in magnetostrictive materials at their inductive excitation by radiofrequency (RF) and magnetic pulses using pulsed NMR and magnetic video-pulse excitation techniques.

Along it the stimulated domain-acoustic echo (DAE), possessing a long-term acoustic memory was inductively excited by three RF pulses in magnetite and magnetoelectric ferrite-piezoelectric layered composites using pulsed NMR technique with inductive and electric recording of DAE signals.

In this work we present also the results of comparative study of different magnetoacoustic responses on excitation by a series of RF and magnetic pulses using pulsed NMR and magnetic video-pulse excitation techniques in some magnetostrictive and magnetoelectric materials such as magnetite, magnetoelectric ferrite-piezoelectric layered composites and magnetic bioceramic composite materials interesting for practical applications in biomedicine. In particular, the magnetic video-pulse excitation technique provides a comparatively simple method for wide-band characterization of these materials.

Keywords

Domain-acoustic Echo; Magnetoacoustics; Magnetostriction; Magnetoelectrics; Pulsed NMR; Magnetic Video-pulse Excitation

Introduction

Different types of magnetoacoustic responses were observed in [12, 9] after the excitation of a magnetostrictive sample mounted in RF coil of a conventional pulsed NMR spectrometer by RF pulses. In [12], a slab shaped ferrite sample was excited by a train of RF pulses of 0.5 microsecond's width and 10 μs repetition time at 5 MHz frequency; and a response signal was observed after each RF pulse. The Fourier transformation of this signal shows a series of equally spaced peaks with spaces depending on sample geometry. Using these data for the slab shape sample, one could find out the acoustic wave speed in this material and compare with similar data obtained from other methods [8]. In [9] the domain-acoustic echo (DAE) signals were inductively generated using pulsed NMR technique after application of three RF pulses in $Co_{0.01}Mn_{0.05}Cu_{0.18}Ni_{0.2}Mg_{0.72}Fe_{0.6}O_4$ ferrite, europium garnet and iron borate samples at 20 MHz frequency.

The acoustic signal $\sigma_1=\sigma_{10}\cos(kx-\omega t)$ (σ_{10}, ω and k are the amplitude, the angular frequency and the propagation constant of the wave, respectively), propagating in a ferrite rod in x-direction after the application of the first RF pulse is recorded applying the second short RF magnetic field pulse $H_1=H_{10}\cos(\omega t)$

of the same frequency ω. As a result of the simultaneous action of acoustic and magnetic pulses, a stationary space-periodic magnetic structure is formed in the sample.

This structure is a magnetic image of the acoustic signal [10, 1]. The formation of this structure could be explained by the irreversible processes of magnetization change. The information storage duration is practically unlimited. The information is read by the third RF or the acoustic pulse $\sigma_2=\sigma_{20}\cos(kx-\omega t)$ as a DAE signal. In case of DAE, the irreversible change in magnetization could be caused by the displacement of domain walls in ferrite grains. In nanosized grains, this change in magnetization is due to magnetostriction [6]. The DAE phenomenon can be used for development of the DAE processors performing integral transformations of RF signals, memory devices, and delay lines [5].

The main properties of DAE were qualitatively well accounted for by a simple phenomenological model [6]. Unfortunately this model cannot be used for the direct quantitative comparison with the experimental results because of its simplicity. So far, polycrystalline ferrites with garnet or spinel structures have generally been used for DAE investigations [7]. The main materials were iron-yttrium garnet $Y_3Fe_5O_{12}$ and nickel ferrite $NiFe_2O_4$. Various kinds of these ferrites ($Y_3Fe_{1.15}Al_{0.85}O_{12}$, $Ni_{0.98}Co_{0.02}Fe_2O_4$, $Ni_{0.97}Co_{0.03}Fe$ and $Ni_{0.97}Cu_{0.03}Fe_2O_4$) were used in DAE experiments. It is seen that in spinels, Fe^{2+} ions are partially substituted by Co^{2+} and Cu^{2+} ions and in garnet, the Fe^{3+} ions are partially substituted by Al^{3+} ones. The Co^{2+} and Cu^{2+} ions in Ni–Co ($Ni_{1-x}Co_xFe_2O_4$) and Ni–Cu ($Ni_{1-x}Cu_xFe_2O_4$) ferrites, respectively, increase magnetoelastic interactions in these materials. Small admixtures of other ions could also change other physical properties of ferrites for optimization of their use in the DAE processors. As an example, small aluminum admixtures in nickel ferrites change their saturation magnetization and a manganese admixture increases their electrical conductivity, while copper one could improve their mechanical properties, etc. As a result, the optimal ferrite composition could become rather complicated.

Additional experimental investigations are necessary to clear out the DAE formation mechanism in magnetostrictive materials with intensive magnetoacoustic responses.

For this purpose, we present here new results on DAE and magnetoacoustic responses study in magnetite, ferrite-piezoelectric (FP) layered composite and a number of other magnetostrictive layered composites, obtained using conventional pulsed NMR spectrometer and its pulsed magnetic field gradient unit [4] for excitation of these samples by three or series of RF pulses and magnetic video-pulses (MVP).

Magnetite (Fe_3O_4), one of the iron oxides, is the most magnetic of all the naturally occurring minerals on the earth. Particles of magnetite are raw materials extensively used for production of magnetic fluids, microwave absorbers, chemical sensors, and so on.

Due to its typical magnetic and electric properties, magnetite is one of the best filler materials used in combination with polymers, e.g. for medical applications and in the information storage media [14].

During the recent years the great attention was paid to study the multilayered FP composites consisting of comparatively thin alternating films of FP materials showing so-called magnetoelectric effect making them perspective for development of new type smart sensors and transducers [13].

In these materials the deformation of the ferrite due to the magnetostriction effect at influence of external ac magnetic field results in the deformation of the piezoelectric layer that is mechanically coupled to the ferrite.

These results in the change in the polarization of the piezoelectric layer were accompanied by the formation of the bound charges on the ferrite-piezoelectric interfaces and, as a result, in the appearance of a voltage U_{ME} generated on the structure surfaces. Such multilayered structures reveal the particular large ME effect which is characterized by a large value of magnetoelectric voltage factor $\alpha_E = \frac{U_{ME}}{h \cdot d}$, where d is the total thickness of piezoelectric layers in the structure and h is the amplitude of ac magnetic field, in contrast with a comparatively weak magnetoelectric effect observed in natural single-phase magnetoelectrics.

In work [3] a comparatively simple method was suggested for wide-band magnetoelectric characterization of a ferrite-piezoelectric multilayer structure using MVP influence. The procedure involves the measurement of magnetoelectric response of a sample to the application of MVP series.

This method allows one to avoid frequency limitations related to the inductance of the coils used to produce the ac magnetic field in methods so far used to study

the magnetoelectric characteristics of samples.

Experimental Results

DAE and Magnetoacoustic Responses in Magnetite and Layered Ferrite-Piezoelectric Composite Generated Using Pulsed NMR Technique

The experimental setup, Fig. 1, used in this work to observe and investigate the DAE and magnetoacoustic signals comprised a standard Bruker Minispec p20 NMR spectrometer for studying the proton relaxation in liquids provided with a Kawasaki Electronic digital signal averager [4]. It was capable to produce 20 MHz RF pulses with durations and powers up to, correspondingly, 10 μs and 100 W. The DAE and magnetoacoustic response signals were observed and studied at room temperature in magnetite samples consisting of disordered naturally occurred magnetite crystalline powders with grain mean diameter ~ 100 μ placed in a cylindrical capsule of about 1 cm^3 volume. Magnetite powder was previously magnetically thoroughly treated to remove the non-magnetic impurities. These signals were also observed under inductive excitation of FP layered composite samples consisting of five slab-shape (10 x 6 x 1 mm^3) layers of commercially available ferrite (F) material used as a ferrite core of television inductive coils and piezoelectric cerium titanium lead ceramics (P), forming PFPFP structure, and electrically detected feeding electric signals from silver contacts connected to the two outer piezolayers to the receiver, Fig. 2.

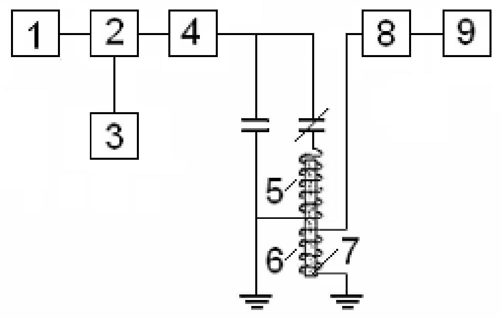

FIG. 1 BLOCK- DIAGRAM OF EXPERIMENTAL INSTALLATION: 1 – RF GENERATOR, 20 MHZ;

2 – GATE; 3 – PULSE GENERATOR; 4 – RF POWER AMPLIFIER; 5 – RECORDING COIL;

6 – READING COIL; 7 – SAMPLE; 8 – RF RECEIVER; 9 – STORAGE OSCILLOSCOPE.

The NMR receiver was locked during action of RF pulse and strong transient response signal of RF coil. Any signal following this moment was absent for the empty coil and for the coil with test manganese and lithium ferrite samples. The signals from samples under study were sufficiently intensive to provide their direct observation on oscilloscope without signal averaging. In addition, intense electric signals from piezoelectric layers were observed which clearly shows the magnetoacoustic nature of the observed signals.

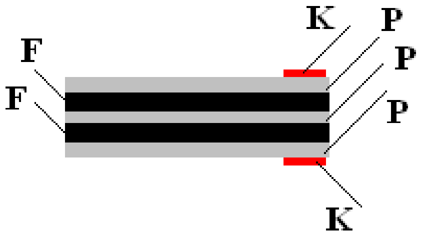

FIG. 2 MAGNETOELECTRIC FERRITE-PIEZOELECTRIC LAYERED PFPFP COMPOSITE:. F – FERRITE, P – PIEZOCRYSTAL; K – SILVER CONTACTS

The FP sample layers were cut using a diamond saw and stacked by epoxy.

Oscillograms of DAE and magnetoacoustic responses in magnetite are presented in Figs. 3a and 3b, correspondingly.

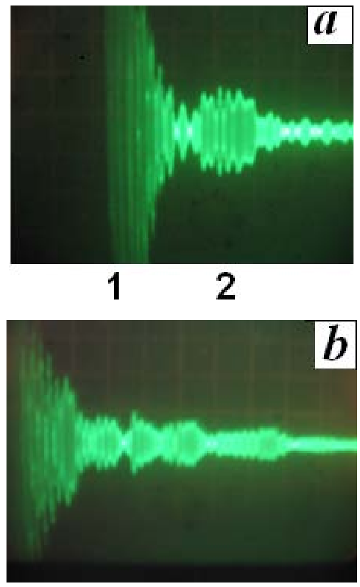

FIG.3. (*a*) OSCILLOGRAM OF DAE SIGNAL IN MAGNETITE. THE TIME INTERVAL BETWEEN THE FIRST TWO RF PULSES: τ_{12}=5 μs, THE RF PULSE DURATIONS τ=5 μs. THE OSCILLOGRAM WAS TAKEN AT ROOM TEMPERATURE, EXTERNAL FIELD H=0 AND 70 W RF PULSE POWER. THE TOTAL OSCILLOSCOPE BEAM SWEEP DURATION IS 100 μs, 1 – THE THIRD RF "READ-OUT" PULSE POSITION MARKED AS 1, THE DAE SIGNAL POSITION – 2. (*b*) OSCILLOGRAM OF MAGNETOACOUSTIC RESPONSE IN MANGANITE UNDER EXCITATION BY A TRAIN OF RF PULSES WITH REPETITION RATE 1 kHz AND RF PULSE DURATION τ=1.7 μs. THE OSCILLOSCOPE BEAM SWEEP DURATION IS 100 μs.

Magnetite DAE dependent on RF pulse power and magnetic field strength are shown in Figs. 4a and 4b, correspondingly.

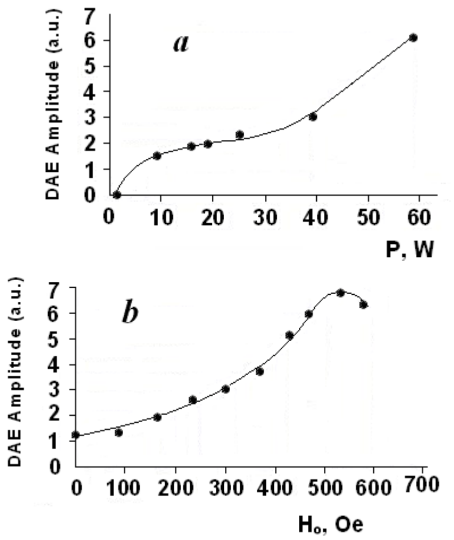

FIG. 4 THE DAE SIGNAL DEPENDENCES ON RF PULSE POWER P AT MAGNETIC FIELD STRENGTH H_O=0 (*a*), AND ON OUTER MAGNETIC FIELD H_O AT P=70 W (*b*) IN MAGNETITE

The strong DAE and ring-down magnetoacoustic signals were also observed in a magnetoelectric ferrite-piezoelectric layered composite PFPFP sample (Figs. 5a and 5b) at its inductive excitation and electric recording of generated signals feeding electric signals from silver contacts K to RF power amplifier.

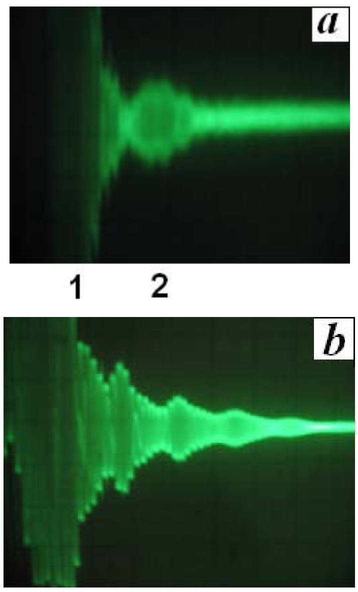

FIG. 5. (*a*) DOMAIN-ACOUSTIC ECHO SIGNAL 2 IN A FERRITE-PIEZOELECTRIC COMPOSITE AT τ_{12} = 5 μs, τ = 5 μs, ZERO OUTER MAGNETIC FIELD, ROOM TEMPERATURE AND RF PULSE POWER P= 70 W. THE END OF RF PULSE IS MARKED AS 1 (*b*) MAGNETOACOUSTIC RING-DOWN SIGNAL AT PERIODIC RF SINGLE-PULSE EXCITATION AT 3×10^3 Hz FREQUENCY AND RF PULSE DURATION 3μs IN A FERRITE-PIEZOELECTRIC COMPOSITE TOTAL BEAM SWEEP DURATION IS 100 μs

Dependences of DAE signal on RF pulse power and magnetic field strength are shown in Figs. 6a and 6b, correspondingly.

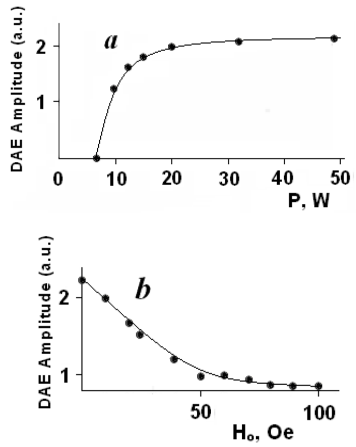

FIG. 6 THE DEPENDENCES OF DAE INTENSITY OF FERRITE-PIEZOELECTRIC LAYERED COMPOSITE PFPFP ON THE RF PULSE POWER P AT H_O=0 (*a*) AND ON THE EXTERNAL MAGNETIC FIELD STRENGTH H_O AT RF PULSE POWER P=70 W (*b*)

It has been established also that the optimal RF pulse powers and magnetic fields for observation of these magnetoacoustic responses coincide with those for DAE signals.

Magnetoacoustic Responses in Magnetoelectric Layered Composites Using Magnetic Video-Pulses Generated By NMR Pulsed Gradient Unit

Other layered magnetostrictive samples were also prepared using similar technique. For investigation of magnetoacoustic responses generated from application of MVPs, it was used current pulses fed through six-turn copper coil supplied by a variable amplitude gated current stabilizer of a NMR pulsed magnetic field gradient unit [4] allowing one to produce the MVPs with amplitude and duration up to 500 Oe and 5 μs, correspondingly.

The experimental setup is shown in Fig. 7 and it is similar to one used in work [3].

In Fig. 8 it is shown an oscillogram of a magnetoelectric response in a layered PFPFP composite obtained using magnetic video-pulse excitation technique at excitation by a series of MVPs.

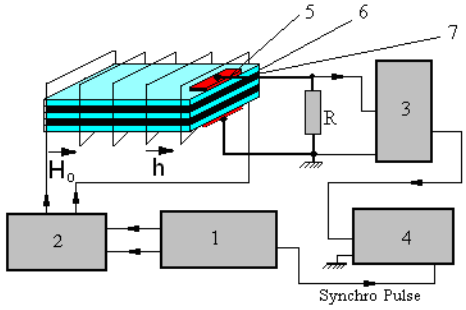

FIG. 7 MAGNETIC VIDEO-PULSE EXCITATION SET-UP: 1. PULSE GENERATOR; 2. CURRENT PULSE AMPLIFIER; 3. VOLTAGE-PULSE AMPLIFIER; 4. OSCILLOSCOPE; 5. SILVER CONTACTS; 6. PIEZOELECTRIC LAYER; 7. FERRITE LAYER

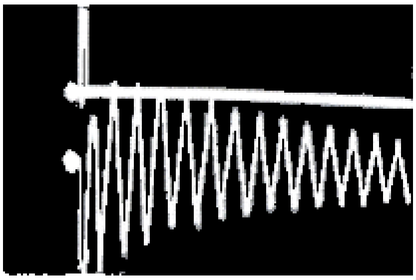

FIG. 8 THE OSCILLOGRAM AT MAGNETOELECTRIC RESPONSE TO EXCITATION BY MAGNETIC VIDEO-PULSE IN A LAYERED PFPFP COMPOSITE. THE UPPER BEAM SHOWS A TIME LOCATION OF MAGNETIC VIDEO-PULSE; THE LOWER BEAM PRESENTS MAGNETOELECTRIC RESPONSE. MAGNETIC VIDEO-PULSE AMPLITUDE H_D=300 Oe, ITS DURATION τ_M=2 μs, REPETITION FREQUENCY 1 kHz. THE FULL OSCILLOSCOPE BEAM SWEEP IS 50 μs

In Fig.9 there presented the results of magnetoelectric response intensity measurements in arbitrary units (a.u.) in a PFPFP composite sample dependent on the value of outer biasing magnetic field (H_o) directed along the surface of the sample at different amplitudes of magnetic video-pulse (H_D).

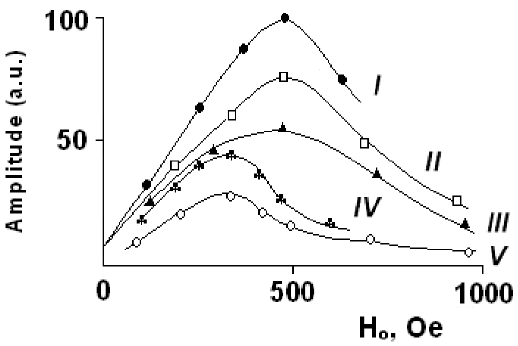

FIG. 9 MAGNETOELECTRIC SIGNAL INTENSITY DEPENDENCE ON BIASING OUTER MAGNETIC FIELD H_O AT DIFFERENT AMPLITUDES OF MAGNETIC VIDEO-PULSE H_D IN PFPFP COMPOSITE. THE DURATION OF MAGNETIC VIDEO-PULSE τ_M =1 μs; ***I***) H_D= 150 Oe; ***II***) H_D= 110 Oe; ***III***) H_D= 75 Oe AND ***IV***) H_D= 37 Oe; ***V***) H_D= 15 Oe

Let us present the results of magnetoelectric response study by MVP excitation technique for two layered composite samples: nickel-piezoelectric composites PNiPNiP in which a commercial nickel foil and piezoelectric layers with thickness 0.15 mm and 1 mm were used respectively to form a stack with dimensions 15 x 10 x 1 mm^3 and the same geometry stack where Ni film was substituted by soft ferromagnetic Co-rich amorphous ribbon Vitrovac 6025 (A) to produce a PAPAP structure layered sample.

The most intensive magnetoelectric responses were generated when the outer magnetic field H_D was directed in perpendicular to the stack surface while MVP directed along it.

In Figs.10 a, b it is presented magnetoelectric responses for this case on outer magnetic field at a definite MVP strength excitation.

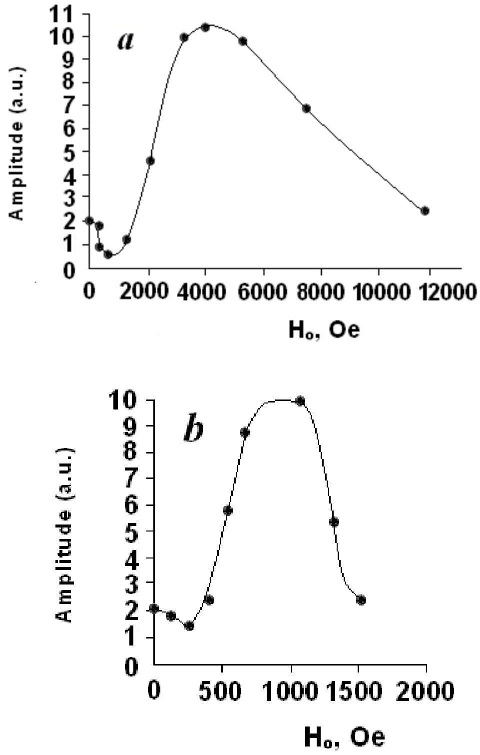

FIG. 10 MAGNETOELECTRIC RESPONSES DEPENDENCES OF THE OUTER MAGNETIC FIELD H_O FOR PNiPNiP LAYERED COMPOSITE (*a*) AND PAPAP (*b*) AT MVP AMPLITUDE H_D = 500 Oe AND ITS DURATION τ_M=3 μs

Similar investigations were also carried out on the magnetoelectric layered composite samples containing the layer of a new magnetostrictive material synthesized on the basis of yttrium phosphate.

Among its precursor components yttrium phosphate (14 %) in the form of a white powder was used as a bonding material. Other components were magnetite and nickel (or cobalt) powders taken at the ratio of 4:1.

The composite material was treated in a laboratory alternative mill during 15 minutes, after which was consolidated in press mould at 130 – 160ºC temperature and 50 MPa pressure during 15 minutes. After the consolidation samples were subjected to thermal treatment in the electric furnace at T=400ºC during 2 hours.

As it is known, the thermal treatment at temperature exceeding 300ºC imparts phosphates with insolubility property in water and improves their mechanical properties.

In Figs. 11a and 11b it is presented the magnetoelectric response signal intensities of the synthesized

phosphate – magnetite composite with Ni and Co commercial powder additives with mean grain size ~ 10 μm, respectively.

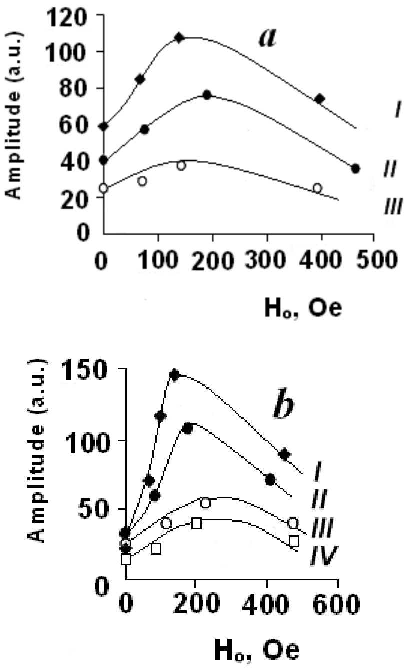

FIG. 11. MAGNETOELECTRIC SIGNAL INTENSITY DEPENDENCE ON THE VALUE OF BIASING OUTER MAGNETIC FIELD H_O AT DIFFERENT VALUES OF MAGNETIC VIDEO-PULSE H_D IN (*a*) PHOSPHATE-MAGNETITE-NICKEL COMPOSITE AT ***I***) H_D = 150 Oe; ***II***) H_D= 375 Oe AND ***III***) H_D= 525 Oe; (*b*) PHOSPHATE-MAGNETITE-COBALT COMPOSITE AT ***I***) H_D = 7.5 Oe; ***II***) H_D= 300 Oe; ***III***) H_D= 450 Oe. THE DURATION OF MAGNETIC VIDEO_PULSE τ_M =1 μs.

Discussion

The basic DAE properties in magnetite are more different than those observed in polycrystalline $Ni_{0.97}Cu_{0.03}Fe_2O_3$ ferrite [10] and for europium garnet in [9] resemble ones of more complicated behavior for iron borate [9]. These peculiarities apparently could be accounted for by the peculiarities of domain wall properties and magnetostriction dependent on the outer magnetic field in this material [12].

It is also noted that the threshold RF power for DAE excitation in magnetite was lower as compared with other studied by us so far magnetostrictive materials.

The first observation of long-term acoustic memory in a layered magnetostrictive polycrystalline ferrite - $LiNbO_3$ medium was made in [2]. In our work the strong DAE signals were inductively excited and electrically detected in a ferrite-piezoelectric layered composite samples using pulsed0 NMR technique.

The DAE signal intensity dependence on outer magnetic field is similar to the one in [2]. The magnetoacoustic responses were also observed in magnetite and ferrite-piezoelectric layered composite samples using the same technique. They have magnetostrictive nature common with DAE signals apparently related to domain wall displacements under the action of RF pulses in magnetostrictive materials.

The studied results of magnetoelectic signals generated from application of magnetic video-pulses in a number of layered composites consisting from alternating layers of magnetostrictive and piezoelectric materials are presented in this work.

In difference to multilayered magnetoelectric samples (NZPO-PZT) studied in work [3], our samples contained smaller number of layers (up to 5) at their larger thickness (up to 1 mm).

Samples in work [3] contained 11 layers of NZPO and 10 layers of PZT at thickness 8 μm each and dimensions 5.9 x 7.3 mm^2. Although values of magnetoelectric voltage U_{ME} and factor α_E in the studied samples were smaller than that in work [3] almost by the order of value, nevertheless magnetoelectric signal U_{ME} in our samples was sufficiently intensive. For this reason and due to the simplicity of their preparation, they could present a practical interest for use in sensors and transducers as well as functional materials – implantants in medicine where bioceramics on phosphate basis are widely used [11].

It is interesting to note that the magnetoelectric response dependent on value of biasing of outer magnetic field observed in phosphate – magnetite-nickel (or cobalt) resembles one observed in this work and in work [3] in samples prepared on the basis of ferrite what could be explained by corresponding change of magnetostrictive factor λ under influence of outer magnetic field in ferrites [13]. Experimental data show strong dependences of magnetoelectric responses of layered samples on their magnetic characteristics, in particular, coercitivity and magnetostrictive factor.

Conclusion

Different long-term acoustic memory and magnetoacoustic effects were observed in magnetostrictive materials at their inductive excitation by RF pulses using pulsed NMR techniques.

Along it the stimulated domain-acoustic echo, possessing a long-term acoustic memory was

inductively excited by three RF pulses in magnetite and magnetoelectric ferrite-piezoelectric layered composites using pulsed NMR technique with inductive and electric recording of DAE signals.

In this work we present also the results of comparative study of different magnetoacoustic responses on excitation by a series of RF and magnetic pulses using pulsed NMR and video-pulse excitation techniques in some magnetostrictive and magnetoelectric materials such as magnetite, magnetoelectric ferrite-piezoelectric layered composites and magnetic bioceramic composite materials interesting for practical applications in biomedicine. In particular, the magnetic video-pulse excitation technique provides a comparatively simple method for wide-band characterization of these materials.

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